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Article

Tensile testing of epoxy-based thermoset system prepared by different methods

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Abstract: Mechanical response of bisphenol-F based epoxy cured with amine hardener was investigated in tensile testing. Different types of methods were considered in preparing the tensile samples in order to evaluate their effects on the tensile strength of the cured epoxy system. Specifically, four types of preparation methods were discussed to prepare the tensile samples were considered in the study. Further, the effect of different type of tensile samples on tensile strength of specimens was also considered in the analysis. The experimental results showed that the preparation methods affected the tensile strength of the specimens. Starting from the experimental results, an appropriate testing methodology is proposed for epoxy based nanocomposite composite specimens in order to reduce problems that may arise during the test and to optimize procedures for preparation of specimens.

Keywords: Epoxy; tensile strength; mechanical properties

1. Introduction

Epoxy resins are broadly used as thermoset coatings, adhesives and matrix in composites mainly in automotive, aerospace, electronics and construction industries due to excellent mechanical and heat resistance properties [1]. During curing process epoxy resin forms three-dimensional structure by crosslinking and obtained cured product is prone to brittle fracture. Numerous approaches are studied and reported to increase the toughness of epoxy thermosets. Mainly the use of rigid fillers mainly (SiO_2 [2], TiO_2 [3], Al_2O_3 [4]), rubber based modifiers [5, 6, 7], block copolymers [3, 8] and MWCNT's [9]. Most of the studies focused mainly on the fracture mechanics properties and thermo-mechanical properties.

Tensile tests are used to measure mechanical properties of materials under tensile loading. The mechanical properties of a polymeric material measured under static tensile loading, are often controlled by chemical bond strength and interaction forces between polymer macromolecules, known as cohesive forces [10]. It depend on the structure of material to be investigated. These forces will rise with the increase in degree of polymerization and average molecular weight. Usually the strength of polymers measured from intermolecular interaction and chemical bonds theoretically is much higher than the values determined experimentally, which may be due the structural heterogeneity. The points of structural discontinuity creates the stress concentration points which leads to final fracture of the material. The tensile test is considered as the fundamental test in material testing among quasi-static testing. Various approaches to perform tensile tests are possible for polymers requiring different loading conditions, different specimens and/or claming devices [11].The molecular structure, crosslink density, testing temperature, and testing rate significantly affect the tensile behaviour of thermoset polymers [12]. The effect of crosslink density on tensile strength, tensile modulus, and elongation at break has been studied by several researchers. The increased crosslink density enhances the tensile strength and tensile modulus but with decrease in elongation at break [13]. While decrease in cross link density by blending DGEBA with aliphatic, cyclo-aliphatic or reactive diluents results in lower tensile strength and tensile modulus [13, 14]. For conventional

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isotropic materials, typical dog-bone specimen geometries are adopted to drive the failure in the waist section under tensile loading [15].

The preparation procedure for the specimens to be tested under static tension is of great significance to the results of investigation. In the literature, to the authors' best knowledge, the effect of the manufacturing process of tensile specimens on the mechanical properties of the specimens was not generally taken into account especially in case of thermosetting materials. However, for epoxy based nanocomposites the preparation method is very significant so as to minimize the bubbles produced during the mixing process at higher filler loading. The present work considers the effect of the different manufacturing process of dog-bone specimens on tensile properties of epoxy based system.

2. Materials and Methods

The EPON™ Resin 862 (diglycidyl ether of bisphenol F) liquid epoxy resin produced by Hexion Inc, low viscosity, liquid epoxy resin manufactured from epichlorohydrin and bisphenol-F is used as a base matrix [16] which has an epoxy equivalent weight (EEW) of 169 g.eq^{-1}. The curing agent was ethacure 100, which is aromatic amine, based curing agent supplied by Albemarle. The resin and hardener was used in the ratio of 100:27.

2.1 Preparation of tensile test samples:

Initially, epoxy resin was preheated in oven at 50 °C to make the handling easier by lowering the viscosity of resin. Then stoichiometric amount of amine hardener was added to the resin and mixed with dissolver aggregate (Dispermat, VMA Getzmann GmbH), then heated up to 55 °C for 20 min. Finally the reactive system was cast in to molds using different methods see Figure 1. The samples were then cured using a three step curing cycle: (1) 90 °C for 4 h, (2) 105 °C for 4 h, and (3) 120 °C for 18 h. For performing the tensile tests DIN EN ISO 527- 2 (type 1B) geometry was selected. Four different methods are discussed here which were used to prepare the tensile test sample. They are as follows:

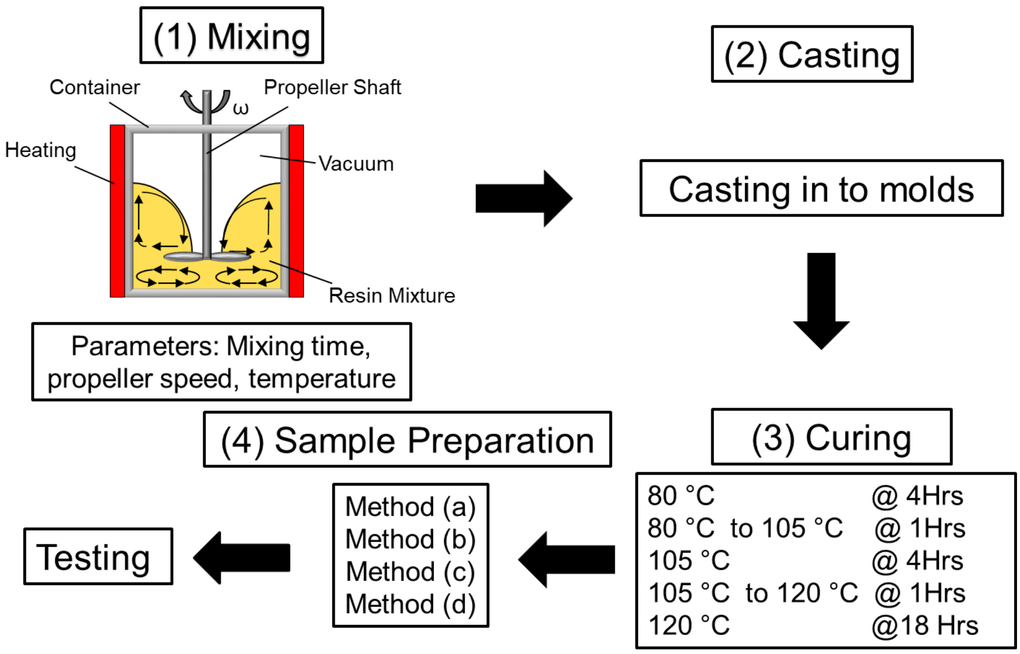

Figure 1: Schematic diagram showing the preparation of epoxy samples through different preparation methods.

2.1.1 Glass mold method

In this method, the epoxy hardener mixture was casted into glass molds, coated with PAT-607/FB (E. und P. Würtz GmbH & Co KG, Germany) release agent and the mold was preheated at 60

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°C for an hour to remove the moisture content and maintain the temperature of mold same as that of pouring mixture. The dimensions of glass plates are 35 cm x 35 cm (see Figure 2a). A glass strip of 4 mm thickness and silicone rubber of 4mm diameter was introduced to between the two glass plates to create a gap and to prevent the epoxy/hardener from leaking. The samples were cured using prescribed curing schedule. In this process, the flat plate of cured epoxy was obtained after the curing. Once flat plate of uniform thickness was obtained then with the help of power hacksaw the rectangular specimens were cut with dimensions of 150 mm in length and 20 mm in width. The obtained rectangular specimens were placed carefully in Mutronic Diadrive 2000 CNC machine (see Figure 3a), maximum of five samples were placed in one run. Once the whole run was completed the dog bone shaped samples were obtained (see Figure 3b). The advantage of using this type of mold is that when it is placed vertically for the curing process all the bubbles moves towards the top surface of the mold and cured plate obtained after the post cure cycle is bubble free and uniform thickness of the samples are obtained.

2.1.2 Steel mold

In this case, the resin hardener mixture was poured in to the mold (see Figure 2b). Prior to pouring the releasing agent was applied gently to the surface and mold was preheated at 60 °C for an hour to remove the moisture content and maintain the temperature of mold same as that of pouring mixture. After the curing process once we have cured plate, then the same procedure was followed as in glass mold method described in section 2.1.1.

2.1.3 Steel molds (with dog bone shaped cavities)

In case of steel molds having dog bone shaped cavities the resin – hardener mixture was directly poured in the cavities carefully. Prior to pouring the was releasing agent was applied gently to the surface and mold was preheated at 60 °C for an hour to remove the moisture content and maintain the temperature of mold same as that of pouring mixture. The mold was then placed in the oven and once the curing cycle was completed the samples were taken out from the steel mold.

2.1.4 Silicone rubber mold

In case of silicone rubber molds having dog bone shaped cavities (see Figure 2d) the resin – hardener mixture was directly poured in the cavities carefully. Prior to pouring the was releasing agent was applied gently to the surface and mold was preheated at 60 °C for an hour to remove the moisture content and maintain the temperature of mold same as that of pouring mixture.

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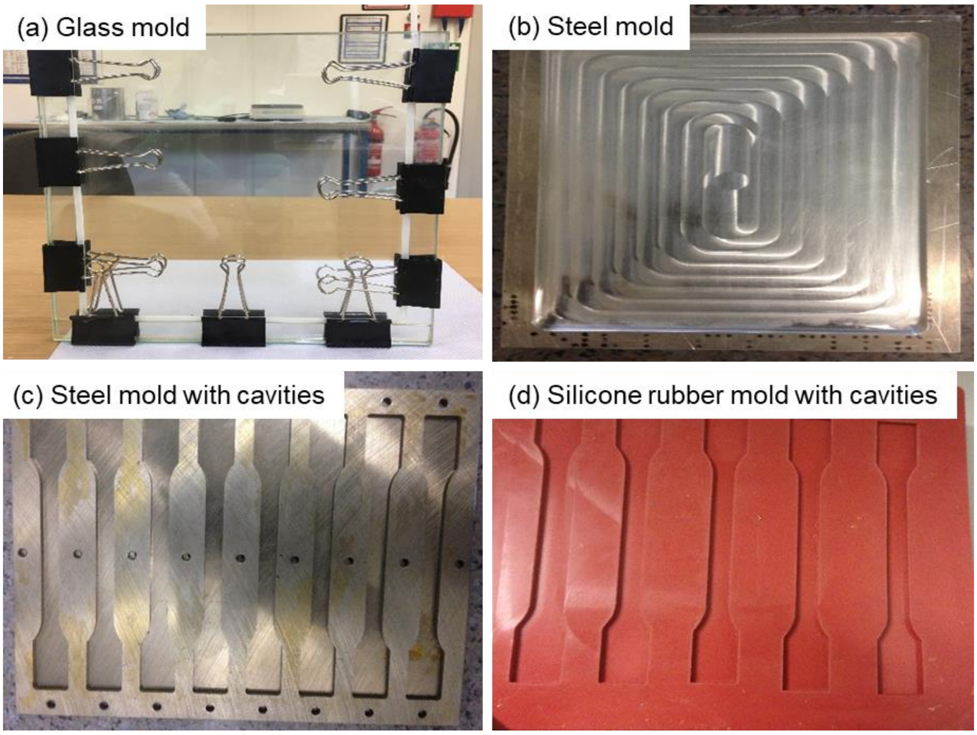

Figure 2: Different methods used for manufacturing of the epoxy samples

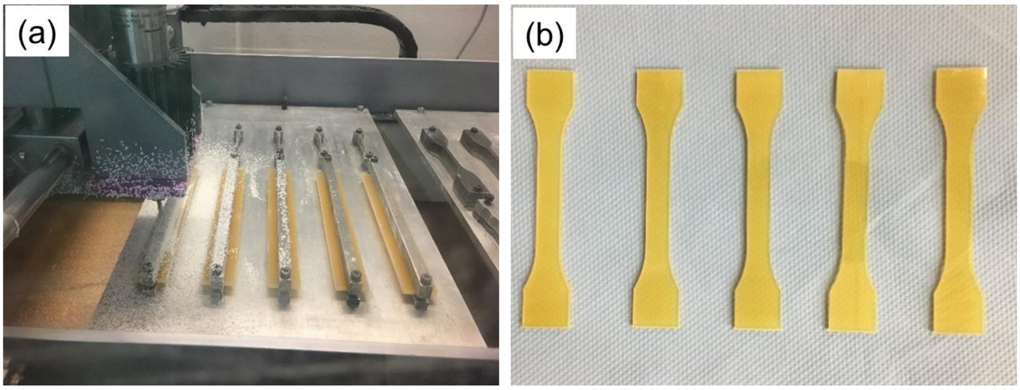

Figure 3: (a) Milling machine cutting the rectangular samples in to dog-bone samples. (b) obtained dog-bone samples obtained after cutting.

2.2 Tensile tests

Tensile tests were conducted at 23 °C on a universal testing machine Zwick 1474, Zwick Roell AG, Ulm, Germany in a tensile configuration as shown in Figure 5 according to standard DIN EN ISO 527-2 [1]. Dog-bone shape (ISO 572-2 type 1B (Figure 4)) samples were used for the testing. Samples have a gauge length of 50 mm, 10 mm width, and thickness of 4 mm. The distance between the sample clamping's was 115 mm and the testing speed was chosen to be 2 mm/min with a 10 kN load cell, a precision sensor-arm extensometer was used to determine the specimen strain. A pneumatic clamp was used to grip the sample. The pressure can be adjusted between 1 bar to 6 bar. However for present study a pressure of 1.5 bar is chosen since at pressure below 1 bar slippage of samples observed at the grip and at pressure above 1.5 bar value cause breakage of samples at the clamps during the tensile test.

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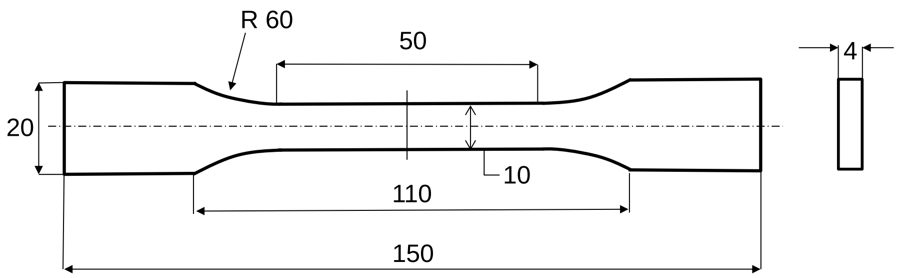

Figure 4: EN ISO 527-2 type 1B geometry for the tensile test specimen [15]. All the dimensions are in mm.

From the resulting stress-strain diagrams, the tensile modulus was determined from the slope of the curve between 0.05 % and 0.25 % of the total strain. The tensile strength was determined from the material maximum sustained stress. A minimum of five samples were tested for each formulation as required [15]

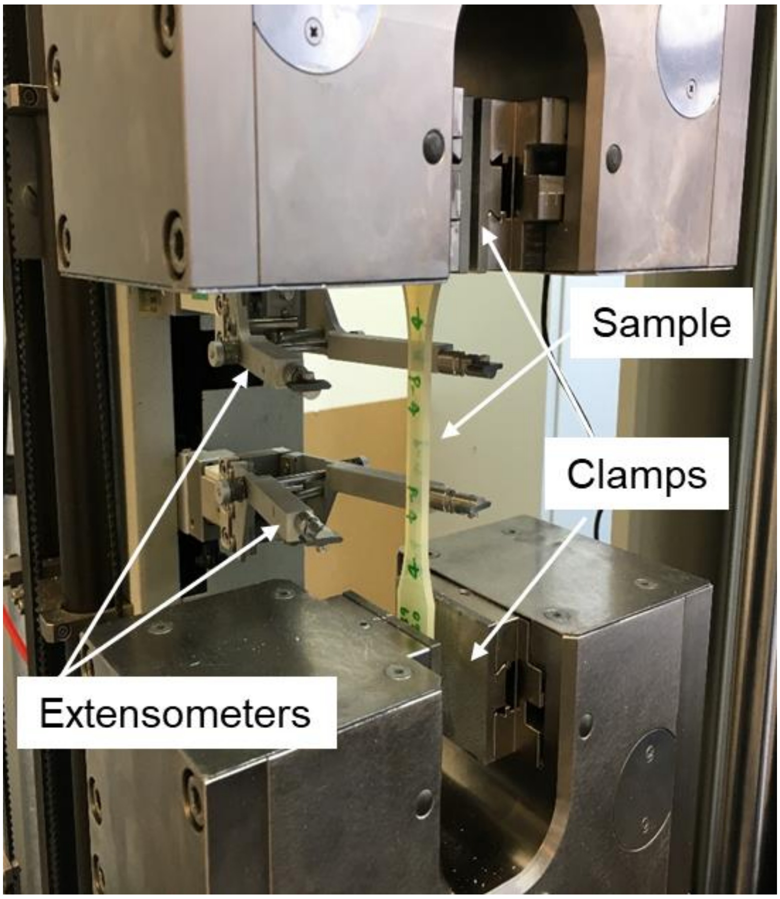

Figure 5 Universal testing machine set-up used for present study.

3. Results and discussion

It can be clearly seen from the table that sample from glass mold have the consistent results as compared to other samples processed through different methods. Main reason behind this trend was the constant sample thickness of the tensile samples and finished side edges, which was achieved through milling process.

In preparation, method (2, 3 and 4) uniform sample thickness was not achieved since one face of the system remained open during the curing process of epoxy-hardener mixture. When rest three methods were compared, the samples produced via method 2 produced better results as compared to method 3 and 4. Reason for these outcomes was lying in the finishing in the edges. In preparation method 2 once the cured plate was obtained the samples were cut in the shape of rectangles (150 mm x 20 mm) later the same procedure was followed as that of preparation method one. It was observed

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that dog bone samples directly obtained from method 3 and 4 have notches at the edges (even though they were removed using the abrasive paper) will cause reduction in the values of tensile strength measurement.

Table 1 Tensile properties of epoxy-amine system obtained from different samples.

Preparation method	System	E_t [MPa]	σ_m [MPa]	ε_m [%]
1. Glass mold	EP_H	2950 (± 14.8)	84.0 (± 0.7)	6.3 (±0.3)
2. Steel mold	EP_H	2980 (± 19.3)	81.0 (± 1.9)	6.1 (±0.5)
3. Steel mold with cavities	EP_H	3000 (± 17.6)	80.0 (± 2.3)	6.1 (±0.4)
4. Silicone rubber mold	EP_H	2950 (± 22.7)	80.6 (± 3.1)	6.1 (±0.6)

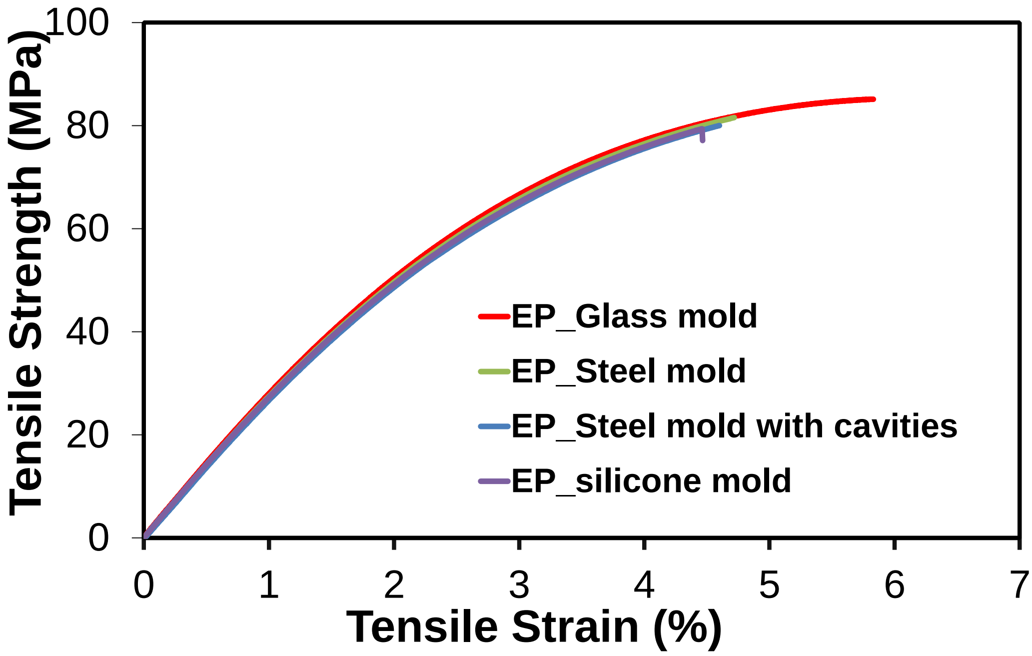

Figure 6 Representative stress-strain curves for epoxy specimens prepared from different methods.

Table 2 Different features observed on tensile samples prepared through different methods.

Preparation method	Uniform thickness	Notches absent at side edges
1. Glass mold	Yes	Yes
2. Steel mold	No	Yes
3. Steel mold with cavities	No	No
4. Silicone rubber mold	No	No

4. Conclusions

Following points can be concluded from the above discussion.

1. Tensile strength depends on the surface finish and the uniform cross-section of the dog-bone samples prepared by different methods.
2. Value of the elastic modulus is independent of the preparation method choosed.

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3. A optimum pressure should be chosen if pneumatic clamps are used so as to avoid slipping of specimens in case of lower pressure in clamps and breakage of specimens in case of higher pressure in clamps.
4. Bubble free cured samples were obtained in case of glass mold method.

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